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Journal of Oil and Fat Industries

Devoted to Oils, Fats and Allied Industries

VOL. I | OCTOBER, 1924 | No. 2

EDITORIALS

SMALLEY FOUNDATION COÖPERATIVE ANALYTICAL PROGRAM

CHECK MEAL SAMPLES FOR 1924–1925

By H. C. Moore, Chairman, Ammonia Committee

The coöperative analytical work, formerly known as "check meal" work, started some years ago by the late Dr. Frank N. Smalley, needs no introduction to most of the members of the American Oil Chemists Society. The possible exception to this may be some of the newly elected members. This work has become of such importance and the results so satisfactory, that in gratitude to its founder, it has been officially named the "Smalley Foundation."

In brief, this collaborative program includes sending out once each week to all members enrolled, a sample of some vegetable meal (last year and the year before, 78 members were enrolled) for determination of Moisture, Ammonia and Oil. The Ammonia determination is made by all collaborators, although not all are interested in the Oil determination.

The order of this work follows a definite schedule. On a fixed date all reports on a given sample must be received by the Chairman, who calculates the accepted average by an established method, and on the following day printed reports are mailed to all collaborators. The form of these printed reports is $3^3/_4'' \times 6^3/_4''$ perforated sheets, the same size as used for Lefax data. Each collaborator is assigned a number and the printed reports do not show the names of the collaborators, simply their number and results.

The series this year starts in September; the report on the first sample must be received by the Chairman by September 22nd. The same grand prize award and certificates of merit will be issued for the best results, as heretofore.

This collaborative work is open to all members of the American Oil Chemists Society. The charge for the full set of samples, which this year may possibly be reduced to 28 or 29 instead of 30 as heretofore, is $15.00.

Federal and State chemists are invited to participate, and will be furnished the samples and reports without charge.

All the members who wish to participate in this work and have not sent in their applications are requested to do so at once, mailing this to Mr. Thomas B. Caldwell, Sec., c/o Law and Co., Wilmington, N. C., accompanied by their check for $15.00.

Any additional information relative to these samples and the work contemplated, will be cheerfully supplied upon request.

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READ THE ADVERTISEMENTS IN THIS ISSUE

The revenue from the advertising section of this JOURNAL will govern the expansion in the section set apart for editorial and original papers.

On the basis of the present membership and rate of growth the income from subscriptions will barely pay for the publication of a 32-page quarterly journal during the next few years. A 32-page quarterly is insufficient even for the publication of the reports and proceedings of the American Oil Chemists' Society. The JOURNAL has a more dignified purpose than that merely of recording the Society's transactions. As it gains in strength and the scientific people recognize it as THE REFERENCE periodical in the English Language covering glycerides chemistry, original work will be drawn to it in considerable volume.

It is not likely that any material changes can be made in the subscription charges. The rates for advertising space have been fixed much lower than experienced advertising people recommended to the Editors. Caution is exercised in extending the advertising privileges in O. & F. I. In presenting to the reader this line of advertising we exhibit the results of a strenuous campaign among worthy establishments having services to offer the oil and fat chemists' plant, library, and laboratory.

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matter." These people make it possible for you to get an enlarged journal at a moderate cost. If the JOURNAL is improved it will be made possible only through the advertisers' support.

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If you are a commercial chemist run your lab card in the DIRECTORY SERVICE section.

The chemists in the American Oil Chemists' Society, by reputation, are the livest bunch among the specialized chemists' bodies. That reputation must be reflected by the JOURNAL. Attention to the little things will insure success in this as in any other establishment. And too, what looks like a little thing may not be, always, so little after all. A little thing can control a big undertaking! And a little help along the lines suggested, from every Chemist-Reader is required if we get the advertisers and build the circulation that furnishes the cash that makes possible the JOURNAL that builds up the Society that works among oil chemists for the better understanding that brings the success that *makes the game worth while!*

F. H. S.

PROCEEDINGS OF THE FIFTEENTH ANNUAL CONVENTION

SAMPLING COMMITTEE REPORT, 1923-24

By P. W. Tompkins

Whatever progress has been made by your Sampling Committee this year, is due to the effort of its individual members and not to the work of its chairman. In the absence of the coördinate effort of occasional committee meetings, it was felt that more ground might be covered to advantage, if the various subjects were made the exclusive business of individuals, as time and efficiency are lost among its scattered members by cross-continent correspondence. In this scheme, there has been some hearty support and it is with pleasure and appreciation that the reports received are submitted for the Society's consideration, as the contribution of its members, to whom all credit is due.

Dr. David Wesson, at his own suggestion, selected the arduous task of developing a bibliography of essential sampling methods, as a basis on which to work toward perfecting any field that future Sampling Committees may determine to follow, and it is hoped that a full measure of appreciation is given this splendid piece of work which is of inestimable and lasting value. Extra copies have been made for submission to your various officers and some means should be taken to make this work a permanent record accessible to all members of the Society.

Dr. Wesson in submitting his report, which is herewith attached, states in substance: That the Bibliography of Sampling was compiled essentially from the *Journal of Society of Chemical Industry*, also from *Chemical Abstracts*, as well as other sources; that quite a number of references have been omitted, which did not seem of particular value in the work with which the Society is interested. His preface to the bibliography mentions that thousands of pages have been written on methods of analysis, as compared with a few hundred on methods of sampling, though it is well known that the first of the three essentials to correct analysis is obtaining a correct sample, and the chairman would like to add that the value of this bibliography will become greater as this field is more fully developed in its many neglected phases.

Mr. G. Worthen Agee, of the Seeds, Hulls and Linters Section, has perfected rules which give evidence of a very intimate and studied knowledge of the subject, and has submitted them in a form immediately available to place before the rules committee for consideration. It would be presumptuous for anyone with less experience in this field to offer any suggestions to what appears to be a thorough conception of the needs.

In submitting his report, which is here attached, Mr. Agee states in substance: That the proposed rules for sampling cottonseed and cottonseed hulls are based on ideas drawn from some of the practical men in the business, from his own engineers who have made a careful study of oil mill problems, and as a result of their own laboratory experience. He further adds that he is making no report on the method of sampling linters, since linters are seldom analyzed in the factory and commercial laboratories connected with the cotton oil industry; that linters sampling of course is of interest to the paper pulp and explosive chemists, but no doubt they have methods of their own for sampling linters, and furthermore that the Interstate Cottonseed Crushers Association has at the present time a special Committee of linter samples and grades.

Mr. H. P. Banks, of the Oil Cake and Meal Section, with his intimate knowledge and experience with Oriental commodities, has furnished a useful contribution of a descriptive character, governing sampling of imported commodities, such as soya bean and linseed cake, fish meal, peanuts, etc. The data furnished in Mr. Banks' report will serve as a basis on which to perfect concrete rules governing these commodities, that future Sampling Committees may wish to cover, which would probably belong to Chapter 16 of the Interstate Cottonseed Crushers Association rules now covering trade in imported oils.

Mr. Thos. C. Law's report, representing the Miscellaneous Section, is not yet at hand, but is hoped will be presented directly in time for consideration by the Association.

Mr. W. D. Richardson, to whom we are looking for a most interesting report on tank car sampling, will probably submit it directly, as it has not yet been received. Mr. Richardson, as Chairman of the A. C. S. Committee on Sampling and Analysis of Commercial Fats and Oils, has the opportunity to coördinate rules which should make them uniform for both organizations.

The chairman feels that a measure of success at least has been obtained by the system of individual sponsorship for definite subjects, and probably as much if not more than would have been accomplished by the older custom.

Committee: P. W. TOMPKINS (Curtis & Tompkins, San Francisco), D. WESSON, W. D. RICHARDSON, H. P. BANKS, G. W. AGEE, T. C. LAW.

Bibliography of Sampling, DAVID WESSON

It has been said that three things are essential to a correct analysis. First, obtaining a correct sample, second, using a correct method of analysis and applying it correctly, and third, drawing the correct reference from the results.

Thousands of pages have been written on methods of analyses as compared with a few hundred on methods of sampling. It has seemed advisable to collect so far as convenient such references to literature of sampling as would be of use to the Committee on Sampling, and to others interested in the subject. References are given and where desirable, brief abstracts or comments are furnished along with them.

J. Soc. Chem. Ind., **2,** 411 (Nov. 29, 1883). "International Methods of Sampling and Testing Commercial Products."—Hurter.

This started a discussion which lasted through the following year. Lunge read report of efforts to secure an international agreement between England, France, Germany and Austria.

J. Soc. Chem. Ind., **3,** 210 (1884).—"Standard Methods of Sampling and Analysis."

Liverpool Section after considerable discussion resolved: That in the opinion of the meeting it would be practicable and very desirable to attain a national agreement on Standard Methods of Sampling ores, raw products, and chemicals, but that an international agreement would be at this time impracticable.

J. Soc. Chem. Ind., **3,** p. 17. "International Methods of Sampling and Testing."—John Pattinson.

J. Soc. Chem. Ind., **3,** p. 339. "On Methods of Sampling."—A. Norman Tate.

Paper before the Liverpool Section goes very fully into the subject in general.

J. Soc. Chem. Ind., **3,** 307 (1884). "On the Practicability of Internationally Establishing and Maintaining Standard Methods of Sampling and Analysis."—Robt. R. Tatlock.

J. Soc. Chem. Ind., **3,** 345–356 (1884). Adjoined discussion on "Standard Methods of Sampling and Analysis and Stating the Results."—Manchester Section.

J. Soc. Chem. Ind., **3,** 650 (1884). "Sampling Apparatus."—H. Augenstein.

German patent 26,680, 1883.

J. Soc. Chem. Ind., **11,** 268; *Eng. Mining J.*, **1892,** 275. "Mixer and Divider for Ore Samples and Small Sampling Machine."—H. L. Bridgerman.

J. Soc. Chem. Ind., **12,** 547; *School of Mines Quarterly*, **13,** 364–368 (1892). "Notes on Sampling."—H. R. Wood.

J. Soc. Chem. Ind., **13,** 214 (1894). "The Sampling of Materials."—T. Clarkson.

A very full paper describing theory of sampling and mechanical appliances to secure accuracy.

J. Soc. Chem. Ind., **13,** 197. "Some Notes on Sampling."—David Sorley.

J. Soc. Chem. Ind., **13,** 277.—"Sampling by Quartation." *J. Am. Chem. Soc.*, **15,** 260–265 (1893).—P. W. Shimer and S. K. Reifsuyder.

J. Soc. Chem. Ind., **17,** 694 (1898), Eng. patent 7021, Mar. 23, 1898. "Improved Apparatus for Taking Samples of Pulverized and Granulated Materials."

J. Soc. Chem. Ind., **21,** 724, *Eng. Mining J.*, **1902,** 73–514. "Sampling—Automatic System of."—P. Johnson.

J. Soc. Chem. Ind., **20,** 992, Eng. patent 14,353, Aug. 10, 1900. "Sampling, Averaging, Mixing, and Storing Material (Portland Cement) in Bulk. Process and Apparatus."—T. A. Edison.

J. Soc. Chem. Ind., **24,** 183 (Feb. 28, 1905). "Standard Methods of Sampling—a Review with Some Suggestions."—Martin L. Griffin.

A careful review of the whole subject.

J. Soc. Chem. Ind., **28,** 194 (1902). Martin L. Griffin goes into the "Mathematics of Sampling" and concludes:

"We may therefore conclude that if a chemist is conversant with the history of the product which he is about to sample and knows how to draw proper samples, the number may be very limited, perhaps 10, not more than 20 or 25. If he lacks the knowledge and ability to do this part of the work, the drawing of almost any number of samples will be untrustworthy."

J. Soc. Chem. Ind., **28,** p. 357. "Sampling Coal—Accuracy in."—E. G. Bailey. Abstract from *J. Ind. Eng. Chem.*, **1909,** pp. 1, 78, 161.

Bailey discusses relation between sizes of sample, size of pieces for each quartering or dividing, there being a direct relation between the size of lumps in the car and the size of particles in the final portion weighed out.

J. Soc. Chem. Ind., **29,** p. 138. "Sampling of Ores—Element of Chance in the."—L. T. Wright; *Mining Magazine*, **3,** 353, 358 (1910).

The author deals mainly with the question of the safe weight in the minimum quantity which shall be truly representative of the whole of the ore and shows relation between size of particles and weight of samples.

J. Soc. Chem. Ind., **30,** 1018 (1911). "Sample—Preparation for Assay."—L. D. Huntoon; *Eng. Min. J.*, **91,** 1249 (1911).

Weights to be taken for different sizes of particles to secure accurate samples—$^1/_2$" 50 lbs.; $^1/_4$" 25; 10" 6 lb.; 20" 3 lb.; 40" 24 oz.; 60" 5 or 6 ozs.

J. Soc. Chem. Ind., **30,** 556 (1911). "Crude Glycerine—Sampling of."—O. Hehner and others.

J. Soc. Chem. Ind., **30,** 1103 (1911). "Directions for Sampling Oil and Natural Gas."—I. C. Allen, U. S. Bureau of Mines, *Tech. Paper*, **1-13,** 3 pp. (1911).

J. Soc. Chem. Ind., **30,** 984. "Gases—Methods U. S. Steel Corporation for Sampling;" *Met. Chem. Eng.*, **9,** 302-306, 356-361 (191.).

J. Soc. Chem. Ind., **30,** 547. "Sampling Ores from Cobalt Mines."—F. W. Pagsley; *Eng. and Min. Journal*, **1911,** 91-776.

J. Soc. Chem. Ind., **30,** 278. "Comparison of Methods of Sampling Wood Pulp."—J. Persoz.

J. Soc. Chem. Ind., **31,** 1011 (1912). "Samples of Gas—Means for Collecting Average."—H. J. Westover, N. Y., U. S. patent 1,037,695, Sept. 3, 1912.

J. Soc. Chem. Ind., **30,** 743. "Air, New Apparatus for Sampling, for Dust."—E. J. Laschinger, *Jour. Chem. Met. and Min. Soc. S. Africa*, **1912,** pp. 443-447.

J. Soc. Chem. Ind., **30,** 927. "Pig Iron—Methods of U. S. Steel Corporation, Chemists' Committee U. S. Steel Corporation."—*Eighth Int. Cong. Applied Chem.*, **1912.**

J. Soc. Chem. Ind., **30,** 928. "Gold Bullion—Sampling of."—F. P. Dewey, *Eighth Int. Cong. Applied Chem.*, **1912.**

J. Soc. Chem. Ind., **30,** 880. "Silver Ores—Sampling and Assaying."—J. O. Handy, *Eighth Int. Cong. Applied Chem.*, **1912.**

J. Soc. Chem. Ind., **32,** 590 (1913). "Sampling Coal Deliveries, Types Govt. Specifications."—G. S. Pope, Bureau of Mines, *Dept. Int. U. S. A.*, Bulletin, 63, 1913, Washington Govt. Printing Office.

J. Soc. Chem. Ind., **32,** 928. "Apparatus for Sampling Fine Powder or Liquids."—T. Roberts.

J. Soc. Chem. Ind., **32,** 1092. "An Automatic Gas Sampling Apparatus with Some Observations on Sampling."—Thomas Gray.

J. Soc. Chem. Ind., **32,** 664. "Plant or Apparatus for Sampling Ores."—A. S. Cleft, London, U. S. patent 1,062,636, May 27, 1913.

J. Soc. Chem. Ind., **32,** 154. "Sampling Sugar Cane Juices."—W. C. Nilboer.

J. Soc. Chem. Ind., **33,** 809 (1914). "Method and Apparatus for Sampling Gases."—E. W. Brown, U. S. patent 1,100,171, Oct. 7, 1912.

J. Soc. Chem. Ind., **32,** 714. "Sampling Liquids—Apparatus for."—R. L. Chambers, English patent 17,828, August 5, 1912.

J. Soc. Chem. Ind., **32,** 905. "Apparatus for Sampling Liquids in Tanks."—E. Schmitz. See *Matiere Grassè*, **7,** 1914–4494 (1914).

J. Soc. Chem. Ind., **34,** 1915.

J. Soc. Chem. Ind., **35,** 896 (1916). "Apparatus for Taking Samples of Flowing Pulp, Slimes, Ores, etc."—P. Davien.

J. Soc. Chem. Ind., **36,** 544 (1917). "Sampling Device for Pulp Digestors."—P. Tuttle.

J. Soc. Chem. Ind., **38,** 59 R (1919). "New Sample Device for Sampling."—L. J. Riley.

J. Soc. Chem. Ind., **39** (1920). Nothing.

J. Soc. Chem. Ind., **40** (1921). Nothing.

J. Soc. Chem. Ind., **41** 611 A (1922). "Sampling Horn."—Kellogg.

New York Produce Exchange has definite rules for sampling materials which are set forth in annual report from July 1, 1921 to July 1, 1922.

Rules and By-Laws of the Chicago Board of Trade give no definite rules as to how samples should be drawn.

Rules of the New Orleans Cotton Exchange do not specify methods of sampling.

Charter, By-Laws and Rules of the Savannah Cotton Exchange do not specify methods of sampling.

Interstate Cottonseed Crushers Association has very full set of the rules for drawing samples. There are opportunities, however, for improving on some of them.

Railroads: Correspondence with a number of railroads indicated in several cases that methods of sampling had never been heard of. On the other hand, The New York Central Railroad Company issued booklet entitled "Instructions Concerning Sampling of Material for Test, May 1, 1917." The Pennsylvania Railroad gives instructions for drawing samples in connection with specification of materials. The same seems to be the case with the Chicago and Northwestern Railway Company.

Specifications for inspection of materials for the Navy Department: General instructions only are given without specifying details as to how sampling should be taken.

General Supply Committee of the Government issue specifications but do not state how materials should be sampled.

Cotton Oil Press, **4,** No. 3, p. 87. "Report of Sub-committee on Sampling Cottonseed—Recommendations for the Sampling of Cottonseed, Peanuts, Soya Beans and Copra."

Cotton Oil Press, **4k,** No. 9, p. 50. "Errors in Official Methods of Sampling Tank Cars."—P. W. Tompkins and E. S. McElligott of Curtis and Tompkins.

Cotton Oil Press, **5,** No. 10, p. 29. "Notes on Sampling Deep-Sea Tanks."—P. W. Tompkins, of Curtis and Tompkins.

Cotton Oil Press, **5,** No. 10, p. 32. "Are Cottonseed Samples Representative?"—A. C. Goetz.

Cotton Oil Press, **6,** No. 1. "Sampling Committee Report, 1921–1922."—M. J. Falkenburg.

Cotton Oil Press, **6,** No. 10, p. 32. "Standard Methods for the Sampling and Analysis of Commercial Soaps and Soap Products."

Cotton Oil Press, **6,** No. 11, p. 33. "Standard Methods for the Sampling and Analysis of Commercial Soaps and Soap Products" (*continued from No. 10*).

R. Dods, *Chem. Eng.*, **11,** 58–68.

J. Soc. Chem. Ind., "Accuracy of Sampling:"

Bailey, **28,** 1924.

Weld, **2,** 426–7 (1883).

Bailey, **2,** 543–4 (1883).

Mining Science Press, **111,** 79–80 (1915). "Relative Error of Sampling."—Haley.

"Ammoniacal Liquid Concentration."—W. C. Klotz, *Chem. Eng.*, **12,** pp. 47–8.

"Notes on Sampling—and Testing."—Anon. Marsden and Co., Manchester, England, pp. 94.

"Sampling and Assay of the Precious Metals."—E. A. Smith. London, C. Griffin and Co. 476 pp., 15 shillings.

"Min. Sampling and Valuing."—Herzig and Purington, San Francisco, Mining and Scientific Press.

"Standard Methods of Sampling for Check Analysis," *Met. Chem. Eng.*, **10,** pp. 701–2.

"Sampling—Churn Drill Prospect Holes."—F. G. Moses. *Eng. Mining J.*, **100,** 301–4 (1915).

"The Value of Coal Mine Sampling."—M. R. Campbell, *Econ. Geol.*, pp. 2–48–57.

"Sampling of Liquids—Specifications for." Report of Sub-Committee of American Soc. for Testing Materials. *Drugs, Oil and Paint*, **1915,** 31–47.

"New Sampling Plant—Hamburg," *Eng. Min. J.*, **100,** 140–1(1915).

"Standard Methods of Sampling and Analysis of Standard Samples."—W. F. Hillebrand, *J. Ind. Eng. Chem.*, **8,** 466–9 (1916).

"Establishment of Uniform Methods of Sampling."—H. W. Wiley, *Report of International Com. on Analysis to the 8th Int. Cong. of Applied Chem.*, pp. 42–69.

"Sampling Apparatus."—30 separate references in Dec. index *Chemical Abstracts* p. 4283 (L–Z).

"Standard Methods of Sampling."—J. R. Barnitz, Chief Chemist General Chemical Co.; "Standard Methods."—Scott, 3rd Edition, vol. II, pp. 1491–1519.

Proposed Changes in Inter-State Rules, G. WORTHEN AGEE

Chapter X—Sampling Article 2—Method of Sampling

RULE 240—COTTONSEED

Section 1. On arrival of car at mill seed shall be first sampled as provided for hereafter in Section 2, and if found off quality shipper shall be notified before unloading but allowance for excess moisture, foreign matter and damaged and immature seed shall be based on test of sample drawn when car is being unloaded, as provided hereafter in Section 3.

Section 2. In Cars before Unloading.—When necessary to sample cars of cottonseed before they are unloaded samples shall be taken in both ends and middle of car at eight or ten different points, not less than fifty pounds in all. As representative samples as possible shall be obtained by digging well down into the body of the seed, preferably not less than thirty (30) inches with a short handled, eight tine fork. The large sample may be reduced in size, if necessary, by carefully quartering and mixing, taking precautions to prevent separating the bolls, trash and sand from the seed.

Section 3. In Cars when Unloading.—Seed can best be sampled at this time. A sack shall be placed in the car and representative portions of the seed placed in it from time to time at such intervals as shall allow of not less than fifty pounds being obtained by the time the car is emptied. This large sample shall be mixed and divided into two twenty-five pound samples, one of which shall be saved for arbitration purposes if so needed.

Section 4. Wagon Seed.—Representative samples shall be taken from a number of points in the wagon, not less than five pounds in all and well down in body of seed. Allowance for off quality shall be based on this sample.

Section 5. Laboratory Samples.—Samples to be sent to a laboratory for analysis of grading shall be about one (1) pound in weight and shall be enclosed in an air-tight container such as a mason jar or friction top metal can to prevent loss of moisture. To insure representative laboratory sample the large sample shall be quartered down as described in the Note appended to the Rule.

For grading, one hundred (100) seeds shall be taken at random from the well mixed sample and shall be cut in two, lengthwise. All seed the kernels of which are darker than the natural color shall be classed as damaged seed; the percentage of damage shall be determined by counting the damaged and immature seed. Determination of moisture shall be made in accordance with the official methods of this Association.

NOTE: The process of quartering a sample to proper size is so important that a detailed description follows:

Mix the large sample carefully and heap it up into a cone shaped pile. Flatten this out into a round flat pile and divide into four equal parts by drawing two lines through the center at right angles to one another. Discard two of the diagonally op-

posite quarters, remix the remaining two and again heap, flatten out, divide and discard two of the quarters. Continue this process until the sample is reduced to a desired size. Care must be observed to see that sand, bolls and other foreign matter are thoroughly incorporated into the seed in mixing

RULE 246—HULLS

Section 1. Hulls Loaded in Car.—Draw representative samples from eight or ten different points in ends and middle of car, getting them from well down in body of the hulls. The samples so obtained shall be ten pounds or more in weight and shall be reduced to a suitable size by repeated mixing and quartering.

Sampling of Oriental Oil Cakes, Etc., H. P. BANKS

The results obtained by the Analyst are worthless if the samples are not truly representative of the material under examination. This statement is axiomatic but it is a cause of continued wonder that so few realize the importance of correct sampling. Preparation of the sample after same is taken is equally important but knowing the origin of the sample brought to the laboratory assists materially. The chemist's section of the *Cotton Oil Press* has contained articles on the preparation of samples of copra, etc., which are valuable.

Our experience with oriental commodities, peculiar to the Pacific Coast, has caused us to establish certain methods for sampling which are generally outlined as follows: though experience and common sense knowledge of the sample is the most important part of the method.

Soya Bean and Linseed Cake

The method of sampling Soya Bean Cake depends upon whether it is "Cartwheel" or "English" style. Cartwheel Cake is sampled by boring the cakes with a 1″ bit. Cakes are selected to represent the pile (as by the Templet Method) and each cake is bored several times, beginning at the center and working toward the outer edge. Due to the variance in moisture content in this type of cake, it is essential that this procedure be followed so that representative sample may be obtained. This type of cake is also subject to mold, and if the shipment should be generally moldy, samples should be taken so that this condition may be noted when samples are brought to the laboratory for analysis and further examination. It is also essential that the sampler examine the shipment for presence of vermin which might seriously affect the merchantability of the shipment.

"English" Style Soya Bean Cake, packed in bags, may be sampled in accordance with Rule 244 of the Interstate Cottonseed Crushers Association.

"Linseed Cake," being in the same form as "English" Style Soya Bean Cake, is sampled in the same manner. However, in sampling Oriental

Linseed Cake it is well to take particular notice to determine whether there is any excess hair on the slabs, this being caused by the seed often being placed in hair cloth bags when pressed. The presence of the hair on the outside of Linseed Cake is detrimental to the quality of the cake. The percentage of cakes bored or bags sampled varies; 5% being the usual accepted standard.

Fish Meal

Fish Meal is sampled by using a trier which is inserted between the twine at the mouth of the bag, a core sample being taken the full length of the bag. Due to the variance in quality, it is customary to take samples in this manner from at least 20% of the bags. Due to the stowage of this material on the smaller vessels, often times Fish Meal arrives in wet and damaged condition. When such is the case it is desirable to make proper segregation when sampling so that the damaged meal might be classified "Fertilizer" while the undamaged meal may rightly be classed as "Edible," if such is the case. The same also applies to shipments which are found to contain mold.

Oriental Seeds

Oriental Seeds of various kinds are sampled by the use of a trier, core samples being taken the entire length of the bag, the percentage of sample taken varying from 5% to 10%. Care should be used in sampling seeds not to make holes in the bags which would cause loss in handling. Linseed is particularly bad in this respect. It is always advisable to use Standard Boerner Sampler to cut down sample to the required size for making purity determination or other tests. Care should also be taken in sampling to take note of condition of shipment as frequently seed, which has not been thoroughly dried, heats in the bags and becomes moldy or otherwise damaged.

Chinese Shelled Peanuts

The sampling of Chinese Shelled Peanuts being mostly hand picked selected nuts differ from most commodities in that samples are taken by hand, opening at least 5% of the bags. Care must be taken in sampling shelled peanuts so that the percentage of split nuts is not increased. Peanuts are very susceptible to heat and moisture and the sampler will do well to take samples next to the burlap as well as into the center of the bags, observing the condition of the nuts when taken from each bag. Samples should also be taken from separate bags so that uniformity of size can be checked when count is made.

In general, the proper sampling of any of these commodities requires that the sampler make a general comprehensive examination of the commodity so that any difference in quality and condition may be reported.

Sampling in the Orient

Conditions in the Orient are so different from those which we find in the United States that the sampler must of necessity make a large part of his inspection while taking the samples. This is due to Customs Regulations and restrictions which are placed upon shipments which are in bond and in transit. Oftentimes Customs Officials will permit the examination of a shipment but will restrict the quantity of sample which may be removed from the shipment. Most of the inspection in the Orient is made in go-downs where the merchandise is being cleaned or graded and prepared for shipment. Under such conditions where there are a number of shipments of the same commodity being prepared, proper markings are very essential.

A considerable volume of business in the Orient is now being done on the basis of Oriental Inspection final which places great responsibility upon the sampler and inspector, one of the requisites being that the shipments in question should be in such condition that they will stand water shipment of from two to six weeks and arrive at destination in Sound Merchantable Condition and of the quality specified in sales contract.

NORWEGIANS ATTEMPT TO REMOVE THE BAD TASTE FROM COD LIVER OIL

From American Consul MAURICE C. PIERCE
Bergen, Norway, Date of Preparation, May 13, 1924.
Date of Mailing, May 13, 1924.

During the past winter Professor Johan Hjort, of the University of Christiania, has carried on extensive experiments for the purpose of removing the disagreeable taste from cod liver oil. He has experimented with more than 500 barrels, which have been steamed and tested in accordance with the method he employs. Good results are now being reported by the local press, which states that Mr. Hjort's experiments have proved that the nasty taste can easily be taken out of the oil, thus enabling it to be used in a variety of food products. Several factories are trying out the tasteless oil in the manufacture of margarine and if the promised results materialize, the United States may lose a group of good customers in Norway. The margarine factories import annually large quantities of cottonseed oil and neutral lard and it is these raw materials that the cod liver oil is intended to substitute.

ORIGINAL CONTRIBUTIONS

STUDIES ON THE OIL AND AMMONIA CONTENT OF COTTONSEED. PROGRESS REPORT ON BASIC RESEARCH PROBLEM I

By A. F. Sievers

One of the research projects suggested by the Committee on Basic Research in Oils and Fats at Washington in October, 1921, was as follows: "To develop varieties of cotton that will yield fibre of maximum value and seed containing the maximum percentage of oil; and also to determine the effects of cultural conditions." This problem was assigned to the Bureau of Plant Industry. The problem of developing varieties of cotton suitable to the several sections of the cotton area has been a major project of the Office of Crop Acclimatization of that Bureau for many years and results on such researches have been published from time to time by Dr. O. F. Cook and his associates. A comprehensive study of the oil content of the cottonseed and its relationship to the many factors involved in the cultivation of this plant is a new undertaking by the Department and it is on the progress of this feature of the basic research problem mentioned that I wish to report to you at this time.

It is probably unnecessary to say that the very nature of the investigation is such that no immediate results can be expected. The work for one season may be very comprehensive and the data obtained quite conclusive, but the fact that we are dealing with a crop that is subject to the influence of climatic and cultural conditions, which may vary from year to year, makes it obvious that the results of a single year or even of several years can be considered only with certain reservations. It is for this reason that in this year's report varieties and stations will not be referred to by name but by symbol and a general summary will be given of the conclusions indicated by the results obtained in so far as they apply to a single year.

The oil content of cottonseed has been the subject of considerable study by some of the Experiment Stations and it is doubtless true that those phases of the problem which concern themselves with the several varieties and with the regional conditions of an individual state can well be undertaken by the Experiment Stations. It is seldom, however, that such a station can conduct its investigations outside the limits of the individual state. The first consideration, therefore, in outlining the program in the Department of Agriculture was to make it interstate in character in the hope that eventually the data thus accumulated might reveal the basic truths regarding the relationship of lint and oil value of cottonseed under the widely divergent conditions obtaining in the entire cotton belt.

It is impossible in a brief report to describe the investigations undertaken at the several Experimental Stations and by other investigators, but it may be well to summarize briefly some of the conclusions drawn from these investigations as obtained from a study of the published reports. It appears to have been concluded that when grown under different conditions certain varieties will rank differently as regards the oil content of their seed; that rainfall has a decided influence on the oil content and that environmental factors such as soil and climate cause a greater difference in the composition of the seed than is due to varietal characters alone. Probably the most important indication is that the greatest single factor affecting the oil content is the maturity of the seed. Immature seed means a lower per cent of kernels and consequently a lower oil content.

In undertaking the study of the seed the Office of Drug, Poisonous and Oil Plant Investigations effected a coöperation with the Office of Crop Acclimatization in order that the latter's field organization might be available for securing the samples. It was arranged therefore, to include samples from a total of 29 varieties grown at 10 different stations as follows: one each in Virginia, Kansas, Oklahoma, New Mexico and Arizona, two in Texas and three in California. The Virginia and Kansas stations represent regions of considerable rainfall; the Texas and Oklahoma stations those of a more limited rainfall, while the far western stations were under irrigation. Although it would have been desirable to include several locations in the southeastern area, time was not available to make the necessary arrangements for last year's season.

The samples of seed were sent to the laboratory from the several stations in friction top tin cans. Some of the samples were sent as seed cotton and were ginned in a hand gin in the Department. The following analyses were made; moisture, average weight of the seed, per cent of kernels, per cent of linters, per cent of oil, and per cent of ammonia. The ammonia determinations were all made by the Barrow-Agee Laboratories at Memphis. Owing to the fact that several sets of samples were ginned by different individuals it was decided at the outset that oil determinations made on the whole seed would probably give unreliable results since it was found that samples from the different stations did not appear to be ginned alike. Consequently the oil determinations were made on naked seed. The available sample of seed was freed from foreign matter and a representative 15 gm. sample selected and delinted with concentrated sulphuric acid. The seed was thoroughly washed to remove the acid and dried for five minutes in a revolving metal cage in a current of air from an electric fan. It was then weighed to determine the per cent of linters and fuzz. The naked seed was then dried moisture-free and again weighed and then extracted with petrolic ether in accordance with the practice in the commercial laboratories. Some question may be raised as to the accuracy of

the sulphuric acid method of delinting the seed but preliminary trials on a uniform lot of seed indicated that with carefully controlled and uniform technique very concordant results can be obtained. Any error resulting from this method would at any rate be much smaller than the error resulting from making the oil calculations on the basis of the whole seed when such seed is not uniformly ginned. All the data are at hand, however, for making the latter calculations if so desired.

It is to be regretted that of the 10 stations only 3 sent samples from the full set of 29 varieties, and from no station were samples obtained from less than 19 varieties. On the other hand, all the stations furnished samples from 8 of the 29 varieties, but from no variety were samples obtained from less than 5 stations. This lack of complete representation of all the varieties from all the stations has greatly complicated the correlation of the data. Comparisons from several angles were necessary in order that conclusions indicated by general and simple averages might be corroborated. The first inspection of the data consisted of arranging the results in a series of 14 tables in each of which from 5 to 10 stations were compared but in which only those varieties were compared which furnished samples from those particular stations. Thus, in one group 24 varieties are compared from stations A, D, F, G, and H. This group represents a comparison of the maximum number of varieties from at least 5 stations. On the other hand in another group only 8 varieties are compared but the comparison covers all the 10 stations. The purpose of these groupings was to avoid comparing averages from several varieties, for example, unless they were represented at the same station. It was found, however, that deductions drawn from such group comparisons were almost in complete harmony with those drawn from the simple averages.

The analyses were studied from the standpoint of the oil content of the moisture-free delinted seed. On this basis it was found that the 29 varieties could be grouped into three groups, representing varieties of high, medium and low oil content, respectively. As an arbitrary division 9 high, 11 medium, and 9 low oil yielding varieties were designated.

A number of interesting facts are revealed by a study of these three groups. It was noted that the average percentage of oil in the delinted seed of the high varieties was 24.31 while that for the low varieties was 22.43, the difference being 1.88 per cent. If the average oil content of only the four highest oil-yielding varieties are compared with the average oil content of the four lowest varieties the difference is 3 per cent instead of 1.88 per cent. If the calculations are made on the basis of the whole seed the difference in the averages are substantially the same. In this connection it may be stated that among all the varieties and at all the stations the highest oil content noted in the delinted moisture-free seed was 31.74 per cent, while the lowest noted was 15.29 per cent. The variety giving

this high yield at one station yielded only 16.01 per cent at another station, this being a difference of almost 100 per cent. On the other hand, the variety giving the lowest yield of the entire list gave at one station a yield of 28.68 per cent. These figures are quoted merely to show the wide range in the oil content of the seed obtained from the several stations under the conditions which prevailed during the past season. It is quite impossible, however, to convey in this report, without lengthy tabulations, the regularity with which the high and low yielding varieties fall into the high and low groups at the several stations.

Other investigators have pointed out that the percentage of kernel in the seed is an important factor in the yield of oil. This is of course to be expected since the oil resides in the kernel. The percentage of kernels is naturally greater in that seed which is partially naked, such as Meade, Pima, and Seabrooke, but when the oil calculations are made on the delinted seed this factor is eliminated. There still remains, however, the effect of the ratio of kernel to hull. A seed with a thick hull or a poorly developed seed yields a lower percentage of kernel than a thin hulled or a well matured seed. It was observed that the seed in the high oil group contains 3 per cent more kernel than the seed in the low oil group.

In order to determine to what extent the relationship of hulls to kernels might be responsible for the grouping of the several varieties into high and low oil yielding seed the kernels or meats from four of the highest and four of the lowest varieties from the several stations were extracted. The results have not been completely correlated but they plainly indicate that, with few exceptions, the kernels from the high oil varieties actually contain more oil than the kernels from the low oil varieties. They show, furthermore, that the seed from those stations where unfavorable conditions prevented full maturity or full development being attained, the low oil content of such seed is due in part to unfilled hulls and in part to less oil in the kernels. The important fact is that at those stations where all the varieties produced plump and fully matured seed the kernels of the seed in the high oil group showed a distinctly higher oil content than those from the low oil group. This is the basis on which all attempts to differentiate between oil values of the seed of the several varieties must rest because it is less subject to modifying factors than any other basis of comparison.

The average weight of the seed, so far as indicated by the data available, appears to be of minor importance. In fact the average weight is slightly higher for the low oil group than for the high oil group. Important consideration must be given here to the lack of uniformity in the ginning which fact probably makes this data of doubtful value except for comparison with the percentages of oil and ammonia when calculated on the whole seed basis.

The relationship of the ammonia content of the seed to its oil content is

of much importance since it is these two constituents which largely measure the commercial value of the seed. The ammonia determinations were made in the Barrow-Agee Laboratories on the whole seed and the percentage calculated on this basis as well as on the delinted seed basis. A study of these percentages showed that the ammonia averages of the varieties in the high oil group and the low oil group are almost exactly the same, namely, 4.40 and 4.41 per cent, respectively, based on the whole seed, and differ only by 0.13 per cent when calculated on the delinted seed basis. From this it would appear that when calculated on the entire seed or on the hulls and kernels (delinted seed) the ammonia percentages do not show any characteristic varietal relationship. It appears, however, that there is a fairly definite relationship between the oil and ammonia content of the delinted seed when compared with reference to the points of production. Thus at one station the seed produced was exceedingly rich in oil (27.82%), but in ammonia it was far below that from the other stations (4.14%). On the other hand, at two other stations the seed had a low oil content (19.96% and 20.02%), but a high ammonia content (5.55% and 5.69%). At the station which furnished seed with the lowest oil content (18.64%) this was not accompanied by a correspondingly high ammonia content (4.78%), but at this particular station the short season did not permit the seed to mature fully, thus no doubt affecting its normal composition. The relationship apparently indicated is in accord with the conclusions of other investigators who pointed out that conditions favoring an increased oil development appear to have a contrary effect on the nitrogenous constituents.

As previously mentioned, samples were secured from 10 stations all but one of which are located west of the Mississippi River. The 5 westernmost stations are under irrigation and therefore under artificial control as far as concerns moisture. In one of these stations, as already stated, the season is somewhat short for cotton and the seed received from this station was decidedly immature, being light in weight, low in percentage of kernels, and very low in oil content. In fact, the wide range in oil content shown by practically all varieties is due to the immaturity of the seed from this station. At two stations located in a long established cotton district the season was unfavorable and the seed from these stations was also of poor quality although this was apparently due not so much to a short season as to unfavorable growing conditions. Another station, located in a district which normally has sufficient rainfall during the growing season but which in 1923 had very little rain in midsummer, also produced seed of inferior quality. By far the best seed from all considerations was secured from one of the irrigated stations. This seed was large, fully developed, and its oil content was so markedly high that it far outranks any other station. While this great difference in the quality of the seed produced at the

several stations suggests interesting possibilities it serves well to emphasize the fact that these investigations must necessarily extend over a period of years in order to observe the effect of the changing seasonal conditions. It is not improbable that results obtained from the present year's crop may result in an entirely different ranking of the several stations. This is especially true of those stations which depend on natural rainfall and where the temperature ranges are wide and the growing season more or less changeable. Perhaps the most interesting fact, however, is that under all the diverse conditions obtaining at the stations represented the relative rank of the varieties as regards yield of oil has, with few exceptions, been the same at all the stations. Certainly this should be sufficient basis to justify a continuation of this work over a sufficient number of years to eliminate, as far as possible, those factors which change from year to year and which are not generally subject to control.

In conclusion let it be emphasized that the foregoing observations are entirely from the standpoint of seed values as viewed by the oil miller. When these investigations have been carried far enough to warrant definite conclusions it will then be necessary to correlate the value of the several varieties from this standpoint with the evaluation of these varieties on the basis of lint production and their general adaptiveness to specific locations in the cotton area in order to determine the true value of such varieties to the grower.

CONTRIBUTION FROM BUREAU OF PLANT INDUSTRY,
U. S. DEPARTMENT OF AGRICULTURE

A PROPOSED SUBSTITUTE FOR THE PRESENT OFFICIAL LYE TABLE OF THE I. C. S. C. A.

BY H. J. MORRISON

The official lye table for maximum amounts allowed in refining crude cotton seed oil by the Rules of the Interstate Cotton Seed Crushers Association is based on the amount of NaOH necessary to neutralize the free fatty acid plus certain excesses.

The latter figure increases abruptly at each multiple of two per cent in free fatty acids.

Plotting these figures necessarily gives a zig-zag or saw tooth curve.

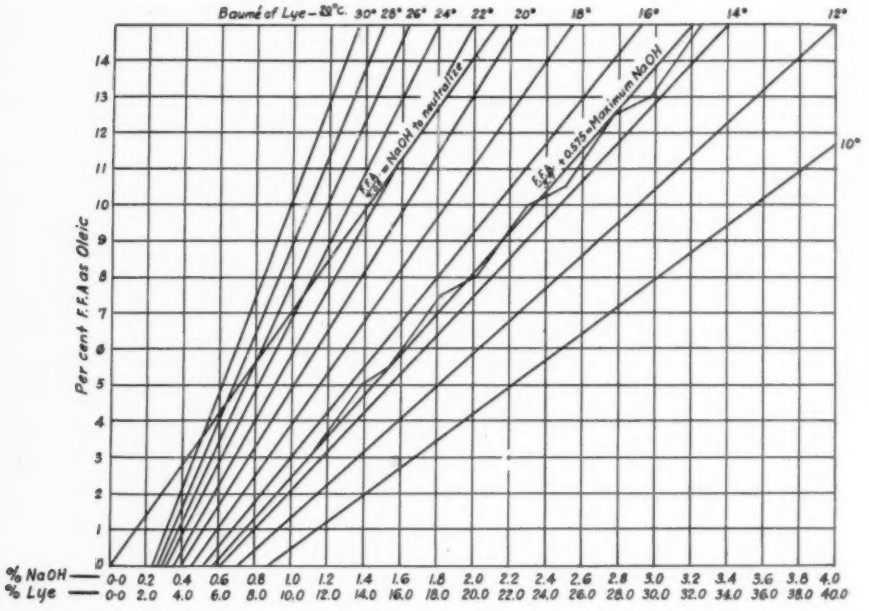
If a straight line is drawn through the average of the saw tooth curve, we necessarily have a curve in which the increases in maximum are gradual and violent differences avoided.

The formula for neutralizing is:

$$\frac{\text{F. F. A.}}{7.05} = \%\ \text{dry NaOH}$$

The excesses allowed in the present official table are experience figures and consist of an increasing excess with increase in free fatty acids.

The chart shows the line of neutralization, the saw tooth line of the present official table and the straight line drawn through the latter.



The formula for this line is:

$$\frac{\text{F. F. A.}}{7.05} + 5.75 + \frac{\text{F. F. A.}}{30} = \frac{\text{F. F. A.}}{5.7} + .575 = \text{Maximum NaOH}$$

It will be seen that this formula consists of the neutralizing factor, a constant excess factor and a factor varying with the F. F. A.

Such a straight line base allows the ready plotting of the various strengths of lye usually used.

These have been plotted on the graph and their convenience for finding the maximum per cent of any strength of lye allowed for a given F. F. A. content is obvious.

The strengths of lye used in these graphs are based on the official Sodium Hydroxide table at 20°C.

At the next convention such a substitute will be submitted to the appropriate committees for action.

THE PROCTER AND GAMBLE CO.,
IVORYDALE, OHIO

STUDIES ON RANCIDITY—A RESUME

By W. C. Powick

Biochemic Division, Bureau of Animal Industry, U. S. Dept. of Agriculture.

Notwithstanding the interest of oil chemists in the subject of rancidity, the chemistry of this condition is still imperfectly known. It has been demonstrated, to be sure, that rancidity is caused by oxidation, presumably by oxidation of the oleic acid radical and of such other unsaturated acids as may be present. It is commonly known that rancid fats respond to general tests for aldehydes and for peroxides, as well as to the Kreis test which is given by some, but not all, aldehydes. Also a few of the constituents of rancid fats have been identified, *viz.*, azelaic acid, and most of the saturated fatty acids and aldehydes up to the C_9 compounds, while heptylic aldehyde has been prominently mentioned as the probable cause of the rancid odor.

Yet very little is known of the chemical mechanism by which rancidity is produced, or of the various factors which might serve to catalyze the process. The exact significance of the Kreis test has been a matter of conjecture and its reliability as an index of rancidity has been questioned because of the positive response that is sometimes obtained when the test is applied to nonrancid cottonseed oils. Even Scala's work, attributing the rancid odor to heptylic aldehyde, naturally involved the personal equation and would seem to be in need of corroboration.

Some of these questions have been discussed and a study of certain aspects of rancidity has been reported in a paper recently published in the Journal of Agricultural Research.* In these studies it was ascertained first of all that oleic acid, even when highly purified, will develop rancidity on exposure to air, responding to the various tests usually associated with the rancid condition; so that the compounds responsible for the odor and reactions of the rancid condition must be among the possible oxidation products of oleic acid. Between twenty and thirty compounds so listed by previous workers, or reported as having been found in rancid fats, were therefore prepared or purchased and submitted to a rapid sorting test. Not one of these compounds responded to the Kreis test for rancidity, while all but nonylic and heptylic aldehydes could be eliminated as sources of the rancid odor. Of these two aldehydes the latter especially resembled rancid fat in odor, although both compounds lacked a certain sharpness of odor which is generally present in rancid fats. Evidently rancid fats contain other constituents—hitherto unrecognized oxidation products of oleic acid—which singly or collectively contribute to the odor of rancidity and are responsible for the reaction of rancid fats in the Kreis test.

* "Compounds Developed in Rancid Fats, with Observations on the Mechanism of their Formation." W. C. Powick, *Journ. Agrl. Res.*, **26**, 323 (1923).

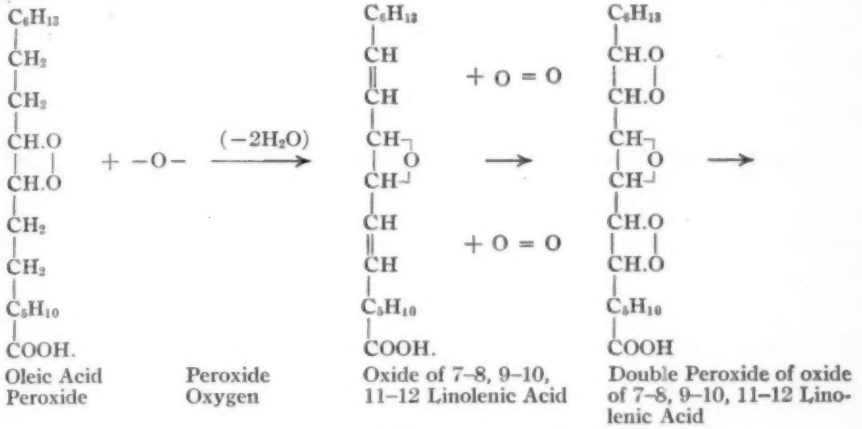
It has long been suspected that the first stage in the atmospheric oxidation of oleic acid consists in the direct addition of molecular oxygen at the double bond with the formation of oleic acid peroxide. This supposition is in harmony with chemical theory and with the presence in rancid fats of peroxide oxygen and of nonylic aldehyde which is presumably formed by cleavage of oleic acid peroxide. A direct study of oleic acid peroxide and its decomposition products would undoubtedly be desirable; but as there is no known method for isolating this compound, the closely related oleic acid ozonide was prepared and examined instead. The latter compound was found to have a sharp, penetrating odor suggestive of that quality in the odor of rancid fats which is not accounted for by the presence of heptylic and nonylic aldehydes; and after partial hydrolysis with formation of nonylic aldehyde, it yielded an odor closely resembling that of rancid fats. Presumably, oleic acid peroxide resembles the ozonide in odor, and is responsible for the sharp quality in the odor of rancid fats. Neither the ozonide itself, however, nor the products obtained by hydrolyzing it with water *i. e.*, the C_9 aldehydes and acids, gave a characteristic response in the Kreis test; but when the ozonide was warmed gently with concentrated hydrochloric acid, evidently experiencing thereby a different kind of decomposition, a definite, though somewhat delayed response, was obtained. Presumably, similar results would have been obtained with the peroxide.

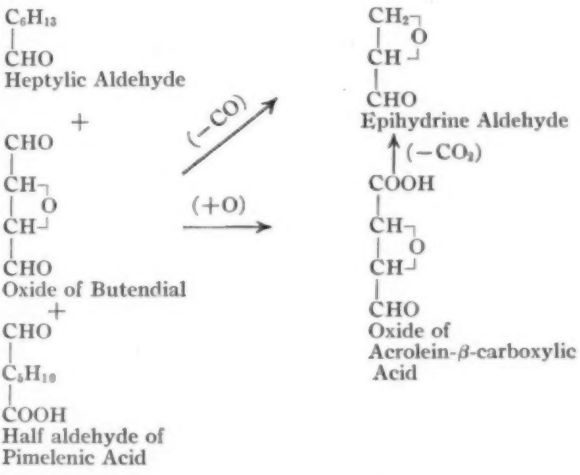
The compound responsible for the Kreis test, however, defied separation and the experiments with oleic acid ozonide offered no clue as to its identity. Attention was therefore directed to the color reactions of phloroglucine. Apparently, most, though not all, of the compounds that yield red condensation products with phloroglucine are aldehydes that contain a double bond—a fact which suggested the examination of acrolein and crotonic aldehyde. In case of acrolein in dilute aqueous solution, no response to the Kreis test could be observed. In case of crotonic aldehyde, on the other hand, an intense red color was immediately obtained; yet this color, unlike that obtained with rancid fats, was fugitive and disappeared within a few minutes when the solution was agitated. The conduct of these aldehydes, when mixed with hydrogen peroxide, however, was entirely different, the acrolein-hydrogen peroxide solution giving an intense and comparatively stable red color in the Kreis test, while the crotonic aldehyde-hydrogen peroxide solution gave no response whatever.

A rigid spectroscopic and spectrophotometric examination identified the Kreis color body obtained from acrolein and hydrogen peroxide with that obtained from rancid fats, and differentiated them both from the similar colors obtained from a variety of other compounds and from certain specimens of nonrancid cottonseed oils. The former colors showed a distinct absorption band in the yellow-green region at wave length 5500 Å, while the latter colors showed continuous absorption at the violet end of the

spectrum. This distinction is probably not without its utility to cotton-oil chemists.

On further investigation of the reaction between acrolein and hydrogen peroxide it was found that the reaction product which gives the characteristic response to the Kreis test is formed only in strongly acid solution, that it reacts with phloroglucine in weakly acid solution, and that it does


Oleic Acid Peroxide
Peroxide Oxygen
(−2H₂O)
Oxide of 7–8, 9–10, 11–12 Linolenic Acid
Double Peroxide of oxide of 7–8, 9–10, 11–12 Linolenic Acid



not contain peroxide oxygen. After examination of a number of the C_3 aldehydes with negative results, epihydrine aldehyde diethyl acetal was synthesized and in the Kreis test was found to yield a red color spectroscopically identical with that obtained from rancid fats and from acrolein hydrogen peroxide mixtures; while elementary analysis of a dried and somewhat altered sample of the Kreis color body prepared from the latter source showed it to correspond in composition to an anhydride of the phlo-

roglucide of epihydrine aldehyde. It thus appears that the color obtained in the Kreis test on rancid fats is due to the formation of a phloroglucide of epihydrine aldehyde.

No direct evidence of the presence of acrolein or of free epihydrine aldehyde in rancid fats could be obtained, although some of the experiments would seem to indicate that the epihydrine aldehyde radical is present in the form of one of its simple compounds, possibly as an acetal, and that its formation is not dependent upon the action of the concentrated hydrochloric acid employed in the Kreis test.

However, that may be, it is certain that the Kreis test depends on the ultimate formation of the epihydrine aldehyde radical, and that the formation of oleic acid perioxide is the first of a series of reactions that may culminate in the production of this radical. The formation of C_9 aldehydes and acids in the development of rancidity is comprehensible enough, but the formation of C_7 (heptylic) and C_3 (epihydrine) aldehydes is somewhat puzzling. Only by the introduction of new double bonds into the oleic acid chain or into that of its C_9 cleavage products, does the further fragmentation of the molecule seem possible. Solway has suggested a plausible mechanism for the formation of heptylic aldehyde and acrolein in the oxidation of linolenic acid; and the accompanying adaptation of his scheme is suggested as a possible explanation of the processes at play in the development of rancidity.

BIOCHEMIC DIVISON, BUREAU OF ANIMAL INDUSTRY,
U. S. DEPT. OF AGRICULTURE

THE SULFURIC INDEX OF COTTONSEED OIL AND IN ADMIXTURES WITH OLIVE OIL

BY JAMES K. MORTON AND G. C. SPENCER, BUREAU OF CHEMISTRY, WASHINGTON, D. C.

Mazzaroni,[1] in determining the thermal index of oils by the Tortelli method, observed that the reaction of the oil and sulfuric acid was always accompanied by the formation of a gas, chiefly sulfurous anhydride, and that the quantity of gas given off varied with the nature of the oil under examination. This led to an attempt to measure the quantity of the gas liberated by passing it into an appropriate iodine solution and noting the quantity of iodine consumed. Mazzaroni termed this factor the "sulfuric index," a heretofore unrecorded constant of an oil.

The purpose of the work here reported was to utilize the principle advanced by Mazzaroni and to show to what extent the sulfuric index can be utilized as a means of identification of simple oils or of the constituent oils in mixtures.

It was necessary to devise an arbitrary procedure and apparatus in order to control every factor that enters into this determination.

[1] *Staz. Sperim. Agrar. Ital.*, **48**, 583–594 (1915).

Oils of low index value, such as olive oil and tea seed oil, offer little difficulty in obtaining a fairly close agreement, even though the oils may differ widely in their source. Oils of high index value, of which cottonseed oil is an example, are sensitive and very wide differences may result with slight changes in apparatus or manipulation. The well-known variable constitution of oils obtained from different sources and the methods of extracting and refining also contribute to variable results.

In order that the sulfuric index of cottonseed oil should be representative of the different grades entering the trade, samples of oil were secured from many of the larger producers throughout the country. The low sulfuric index value of olive oil, which is subject to slight variations, has eliminated, for the purpose of this work, the necessity of extended research on this oil.

Apparatus

The apparatus (Fig. 1) consists of a Pyrex test-tube, 10 by $1^1/_2$ inches. The rounded bottom of this tube must be as nearly hemispherical as possible. A glass stirring paddle the blades of which are 3 cm. from tip to

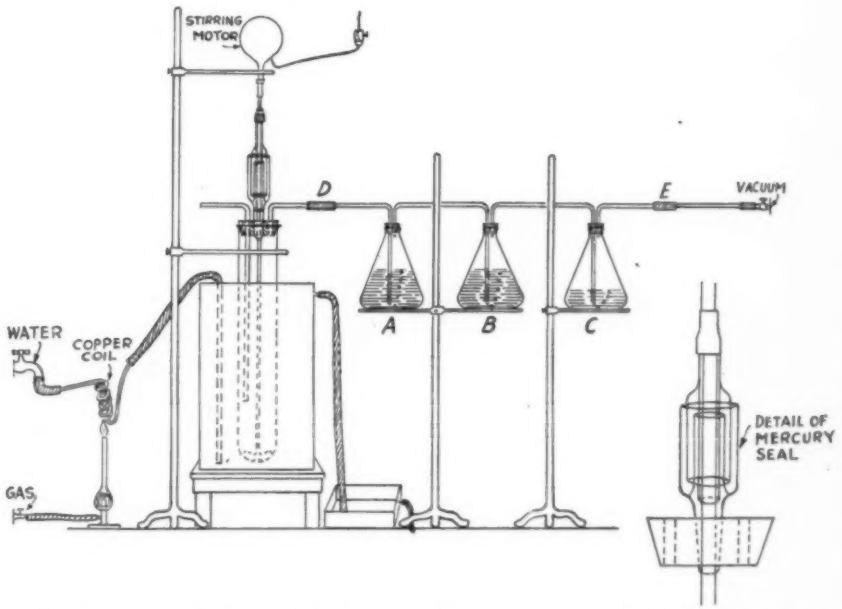


Fig. 1.—Apparatus set-up for the determination of the sulphuric index of oils.

tip and 1 cm. deep, is provided. The blades conform in shape to the rounded bottom of the tube and are turned to give, as nearly as possible, an upward movement to the liquid. A three-hole rubber stopper closes the

tube, the center hole being large enough to admit a mercury seal, the other two holes carrying glass tubes of 4 mm. inside diameter. The shaft of the glass paddle passes through the mercury seal and is attached to the shaft of a stirring motor regulated to run at 300 r.p.m. The absorption end of the apparatus consists of three 200 cc. Erlenmeyer flasks each provided with a two-hole rubber stopper and glass connecting tubes. Glass tubing of 4 mm. inside diameter is used throughout.

The reaction is conducted in a constant temperature bath maintained at $25^1/_2$ to 26°C. This bath can readily be constructed from an empty ether can. A $^3/_8$-inch overflow is provided about an inch from the rim.

Running tap water is passed into the can through a quarter-inch copper coil heated with a Bunsen flame, and the temperature of the bath is controlled by the rate of flow of the water and the heat applied. The authors have found this to be a very efficient and convenient means of bath control the variation not exceeding 0.5°C. It has the advantage of being portable and easily manipulated. The test-tube can be so adjusted that tilting alone is sufficient to remove the bath without disturbing the apparatus above it. The overflow of the bath can serve as an additional bath in which the sulfuric acid and oil under examination may be brought to a uniform and constant temperature before making the determination.

The apparatus is assembled as shown in Figure 1. The intake tube in the digestion chamber extends to within $1^3/_4$ inches of the bottom of the digestion chamber. The intake tubes of the absorption flasks should extend to within a quarter of an inch of the bottom of the Erlenmeyer flasks, the outflow tubes in each case extending flush with the bottom of the rubber stopper. The apparatus should be adjusted so the the stirring paddle when in place will just clear the bottom of the tube and revolve freely.

Reagents

(1) Sulfuric acid (98.5%) prepared by boiling the ordinary 95% acid in an open casserole to about two-thirds of its original volume. It is poured hot into a Pyrex Erlenmeyer flask and cooled under a calcium chloride tube. This acid must be carefully made to a uniform and definite strength and kept well protected.

(2) Iodine, tenth-normal solution.

(3) Sodium thiosulfate, tenth-normal solution.

(4) Starch, 0.5 per cent solution.

(5) Mercury.

Determination

Measure into the first 200cc. Erlenmeyer flask (A) enough tenth-normal iodine solution to assure an excess of about 15 cc.[2] and bring the volume to about 150 cc. with

[2] The quantity of iodine required is indicated by the character of the oil and preliminary test.

water. To the second flask (B) add 5 cc. of the tenth-normal iodine solution and bring the volume to about 150 cc. To the third flask (C) add 1 gram of potassium iodide and 2 cc. of starch solution and dilute to about 100 cc. Connect the three flasks as indicated in Figure 1. Aspirate through the apparatus and regulate the rate to about 180 bubbles a minute.

Bring the sample of oil and the sulfuric acid to 25.5° to 26°C. in the constant temperature bath. Measure with a pipette 20 cc. of the oil and introduce it into the digestion chamber by placing the tip of the pipette near the bottom and touching the side of the tube. Be sure that no oil touches the side of the tube above the level that will be reached when the sulfuric acid is added. Allow the pipette to drain for 10 minutes. Carefully pipette 5 cc. of the sulfuric acid into the test-tube by placing the tip of the pipette just above the level of the oil, touching the side of the tube, and allow the acid to underlie the oil with as little disturbance as possible. All premature mixing must be avoided. Connect the test-tube with the stirring apparatus but do not let the paddle dip into the mixture.[3] Place the constant temperature bath directly under the test-tube. Raise the test-tube to its proper position and fasten in place. Make the connection to the absorption flasks at D. Elevate the constant temperature bath to submerge about 6 inches of the test-tube. Disconnect the aspirating tube at E.[4] Place a finger over the open end of the intake tube and start the stirrer. Steady the digestion tube with the hand to eliminate friction as much as possible. When the reaction subsides, as indicated by the backing up of the solution in the first flask release the finger from the intake tube, reattach the aspirating tube at E, and continue the aspiration at the predetermined rate. Stir for exactly 10 minutes. At the end of 30 minutes disconnect the absorption flasks at D and titrate the excess of iodine with tenth-normal thio-sulfate. The number of cubic centimeters of tenth-normal iodine consumed is the sulfuric index of the oil.

TABLE 1

SULFURIC INDEX OF COTTONSEED OIL

Source	Grade	Deter.	Max.	Min.	Ave.	Iodine number (Hanus)*
Miss.	Prime summer yellow	3	96.1	85.3	89.0	111.1
Ga.	Summer yellow	3	87.0	83.7	84.9	108.9
Tenn.	Prime summer yellow	3	96.2	91.0	92.7	111.6
Tenn.	Refined	2	85.0	74.5	79.7	111.0
Texas	Refined	4	56.37	51.50	53.8	104.0
Texas	Refined, winter pressed	3	69.0	66.7	68.1	102.0
Okla.	Refined	3	65.6	60.0	63.6	104.7
Okla.	Salad	2	79.67	76.9	78.3	106.5
N. Carol.	Refined	2	71.6	67.1	69.3	107.9
N. Carol.	Refined	4	88.0	70.7	81.4	112.2
	Cooking	2	56.2	56.0	56.1	104.0
	Salad	3	64.0	62.0	62.7	107.5
	Butter	5	68.1	56.0	62.6	106.1
	Winter oil	4	88.5	73.7	80.1	108.8

* Iodine numbers were determined by Walter F. Baughman, Oil, Fat, and Wax Laboratory, Bureau of Chemistry, Washington, D. C.

[3] Care must be taken at this point to see that the paddle and test-tube are in correct alignment, permitting free movement of the paddle.

[4] Before starting the motor the aspirating tube should be disconnected from the end flask to permit the escape of air caused by the reaction in the digestion chamber.

TABLE II

SULFURIC INDEX OF MIXTURES OF COTTONSEED OIL AND OLIVE OIL

Sulfuric index of cottonseed oil	Per cent of olive oil added							
	10	20	30	40	50	60	75	100
86.6	60.7	42.0	26.0	17.2	11.5	6.7	3.2	2.54
	57.1	40.5					3.1	2.60
	59.1	39.0						
Iodine No.								
108.3	(105.5	102.8	100.0	98.2	96.5	93.7	89.6)	81.3

(Iodine value interpolated)

For comparison the iodine value is tabulated with the sulfuric index. The iodine value of cottonseed oil as recorded in chemical literature has a range of about 10 units. The sulfuric index range as found in this investigation is about 40 units (Table I). Hence the sulfuric index is more sensitive than the iodine number and close duplication of results will be more difficult.

In mixtures of olive oil and cottonseed oil the sulfuric index is not proportional to the quantity of either oil present. The addition of olive oil to cottonseed oil causes a marked reduction of the sulfuric index out of all proportion to the quantity added. Thus olive oil with additions of cottonseed up to 25 per cent shows but a slight increase in the sulfuric index, while cottonseed oil with 10 per cent of olive oil added shows a marked decrease in the sulfuric index.

It is believed that the addition of a low-index oil to a high-index oil not only reduces this index but also interferes with the heat of reaction of the high-index oil, resulting in a decreased proportional index of the higher oil.

TABLE III

SULFURIC INDEX OF VARIOUS OILS

Oil	Sulfuric index	Iodine value
Olive, California.....	2.1	
	2.54	
	2.60	
	2.2	81.3
Italian.....	1.00	82.2
	1.21	80.0
Tea seed.....	1.62	83.0
Coconut.....	0.80	6.9
Sesame.....	52.67	
	51.97	109.1
Apricot.....	15.1	
	14.3	107.1
Peanut, cold pressed..	5.73	97.1
Sunflower seed	138.1	130.8

The sulfuric index value of a number of other oils was determined by this method and the results are tabulated in Table III.

The sulfuric index has a place in the record of analysis of cottonseed oil. The conditions entering into this determination can be controlled, but if the results are to have any value the method must be followed with care and expert manipulation. As a routine method it does not compare with that for the iodine number in the number of determinations that can be made in a given time, but as a means of identification of an unadulterated oil it compares favorably. The extent of adulteration of cottonseed oil with olive oil may be determined within a definite limit, but the reverse is not true. A large proportion of cottonseed oil can be added to olive oil, without increasing the index measurably.

Acknowledgment is made of the assistance given by Dr. G. S. Jamieson of the Oil, Fat and Wax Laboratory of the Bureau of Chemistry in securing for this investigation the oils under examination and for advice and comment.

BLEACHING STUDIES ON COTTONSEED OIL

By Frank C. Vilbrandt and Herman J. Bankston, Jr.

The production of a light, clear oil from dark-colored crude cottonseed oil is a difficult industrial undertaking and much time and money has been spent in this study. After the process of dragging down the fine meats and the proteins with soap formed in the oil, which in addition reduces the fatty acid content of the oil, many substances have been suggested for further bleaching the oil, but Fuller's Earth or certain clays are commonly used for this purpose.

There are many grades of this earth as well as many grades of oil to be bleached, some of the latter not responding to this treatment at all. Therefore many investigations have been made to determine whether the variability of the composition or of the procedure is responsible for the variations in results. Some emphasis has been laid upon the effects produced by these earths being dependent upon the quality of the earth, the quality of the oil and the skill of the workman in carrying on the bleaching operations. However, studies carried out by the Bleach Test and Fuller's Earth Committee, A. O. C. S., and embodied in their report[1] of August, 1921, indicated that the time and degree of agitation had no influence of the degree of bleach, that it was unnecessary to fix a standard procedure and the skill of the workman was not such an important matter in the bleaching of the oil.

Chemical analyses seem to throw no definite light on the mechanism of the bleach, for bleaches of the same formula often have different bleaching properties, while others with widely different composition have almost

[1] *Cotton Oil Press*, V, 122 (1921).

identical bleach qualities. Oils are encountered that are not affected by increasing the amount of the Fuller's Earth and the greater the amount of earth used, the greater the introduction of foreign tastes and odors imparted to the oil by the Earth.

Therefore the only practical method of gaging the bleaching power of an oil or the bleaching power of an earth sample is by trying it out, and standardizing against known earth and oil sample.

A review of the different methods of bleaching, suggested to the writers, that there may be some chemical reason for the difference in the bleaches. Holbrook[2] used sodium silicate after the reduction of the free fatty acid content of the oil and the settling out of the soap. This silicate, being alkaline, indicates the probability that the coloring matter is acidic and is neutralized by the sodium silicate or precipitated in this alkaline medium. On the other hand, Baskerville[3] uses a patented inorganic acid solvent that he claims has the properties of Fuller's Earth. The use of sulfuric acid is also known, it being supposed that the acid chars or dehydrates the coloring matter and causes it to become insoluble. Many active bleaching Fuller's Earth samples exhibit the property of giving an acid reaction to water in which they are suspended with phenolphthalein as indicator.

This investigation was undertaken with the purpose of finding out whether the acidity or alkalinity of the solutions or the Earth affected the bleaching property.

Bleaching Agents.—The substances that were used for bleaches on the treated oil were animal charcoal, bone black, Georgia clay, China clay, kaolin, Fuller's Earth, Portland cement and coal clinker. The animal charcoal and bone black were ordinary laboratory stock materials; the Georgia clay was taken from the hillside of the campus of Emory University, Emory, Georgia. The China clay and kaolin were also common chemical stockroom material. The Fuller's Earths used were specially purchased products. The Portland cement used was a common brand of construction material, while the coal clinker was taken from a power plant. Only the clinker was given any mechanical treatment previous to its use; in this case the clinker was ground to pass an 80 mesh screen.

Oil Stock.—Five gallons of fresh raw cottonseed oil was obtained from a press that was operating on fresh, new seeds of the season, the fresh oil being of not a bad color with a small amount of the mucilaginous, fine, mealy matter and some tarry substance. The oil had a fatty acid value of 1.8% and a saponification value of 198 (Hanus Iodine Method).

Since all the samples were to be run for relative bleaching activity, a large portion of the raw oil was treated with caustic and clarified, this clarified and washed sample being used as the standard oil to which all

[2] *J. S. C. I.*, XXXV, 317 (1916).
[3] *J. S. C. I.*, XXXIII, 930 (1914).

the different bleaches were added. The factor of variability in the caustic treatment was thereby eliminated from this study, or reduced to the minimum. The weighed oil was heated to 30°C. and a quantity of caustic soda solution slightly in excess of the amount required to take care of the free fatty acids was stirred in as rapidly as possible, and the stirring continued for ten minutes with an electric agitator, during which time the temperature of the mass was slowly raised to 52°C. The agitation was continued until floccing occurred and after settling for five hours the clear, but colored, oil was separated from the settlings. This oil was then washed free from caustic alkali with water, maintaining a temperature of 40°C. throughout. After standing several hours the clearer oil was poured off from the watery emulsion and treated with Plaster of Paris to take out the last of the water. The resultant oil constituted the raw material for the bleaching experiments carried out in this study.

Color Comparison.—The suggested practice of color standard oils for bleaching qualities, that of using a specific English Fuller's Earth was not followed. In its place was substituted the easily reproducible chemical color comparator. This color comparator consisted of 400cc. Pyrex beakers of uniform dimensions, placed on an opaque, white glass, and the reproducible K_2CrO_4 color solutions of the following compositions placed therein: 1% K_2CrO_4 solution, neutral, constituted color A, 5% B, 10% C, 15% D, 20% E, 1% K_2CrO_4, with 5 drops conc. HCl per 250 cc. solution color F, and 5% K_2CrO_4, with 10 drops conc. HCl per 250 cc. G.

All color comparisons were made in the same size beakers, at the same time, and using the same volume of oils and color standards.

Bleaching Results.—The general procedure for bleaching was carried out as follows: Three hundred cubic centimeter portions of the oil stock were heated to 50°C. and at this temperature a definite weight of the bleaching material was added and the mixture agitated 20 minutes. The mass was then filtered through filter cloth on a Buchner funnel. The original unbleached, causticized and washed stock gave a color G, which was slightly darker than the crude oil. A sample of salad oil obtained on the market had a color between B and C, while a marketed cooking oil, summer stock, had a color B.

A preliminary run was made to determine the minimum amount of clay that could be used with the above oil and the above treatment to give the best color. Runs using 2%, 3%, 5% and 10% of Fuller's Earth were made. Results with the use of 5% and 10% were the same, color E; 3% gave color between E and F. Therefore the standard quantity of bleach used to reduce the quantity factor to the minimum in this study was adopted as 5%.

Table 1 sums up the results, giving also the conditions under which the bleaching materials were treated before use.

TABLE 1

BLEACHING PROPERTIES OF COMMON OIL BLEACHES

Experiment No.	Bleaching agent	Condition of bleach	Color comparison results	Troubles in filtering
A	ORIGINAL CRUDE OIL	Raw, none	F-G	
B	Raw treated oil stock		G	
1	Fuller's Earth, No. 1	Raw, untreated	F	None
2	Fuller's Earth, No. 1	Heat 1 hour at 105°C.	E	None
3	Fuller's Earth, No. 1	Heated 1 hr. over Meker burner	D	None
4	Fuller's Earth, No. 1	Wetted, then dried at 105°C.	D	None
5	Fuller's Earth, No. 2	Raw, untreated	F	None
6	Fuller's Earth, No. 2	One hr. drying at 105°C.	E	None
7	Kaolin	Raw, untreated	G	Slight (small cloudiness remained)
8	Kaolin	Heated 1 hr. over Meker burner	C	Slight, (slight cloudiness remained)
9	China Clay	Raw, untreated	G	Difficult to filter
10	China Clay	Heated 1 hr. over Meker burner	F	Difficult to filter
11	Animal Charcoal	Raw, untreated	GG	Could not filter out the char
12	Animal Charcoal	Heat 1 hr. over burner	GG	Could not filter out the char
13	Boneblack	Raw, untreated	GG	Could not filter out the char
14	Boneblack	Heated 1 hr. over burner	GG	Could not filter out the char
15	Clinker	Ground original clinker	GG	Could not filter out the char
16	Clinker	Heated 1 hr. over Meker burner	GG	Could not filter out the char
17	Portland Cement	Raw, stock	G	None
18	Portland Cement	Heated 1 hr. over Meker burner	G	None
19	Georgia Clay	Air dried	E	None
20	Georgia Clay	Dried one hour at 105°C.	F	None
21	Georgia Clay	Heated 1 hr. over Meker burner	B	None
22	Georgia Clay	Same as 21 only used 3% of clay	C	None
23	Georgia Clay	Saturated with concn. HCl, washed 6 times, then dried at 105°C.	G	None
24	Georgia Clay	Saturated with 10% HCl, washed 6 times, then dried at 105°C.	D	None

Experiment No.	Bleaching agent	Condition of bleach	Color comparison results	Troubles in filtering
25	Georgia Clay	Saturated with 10% HCl, washed 6 times, then heated over Meker burner	C	None
26	Georgia Clay	Saturated with 10% NaOH, washed 6 times, then dried at 105°C.	G	None
27	Georgia Clay	Saturated with 10% NaOH, washed 6 times, then heated 1 hr. over Meker burner	F	None

DISCUSSION: It is seen that the clinker, China clay, kaolin, cement, bone black and animal charcoal are unsuitable under the conditions described above because of the difficulty in separating the bleach from the oil. Of these substances only China clay and kaolin after heat treatment have any bleaching properties. The bleaching results one expects with bonechar and charcoal were not present or at least not apparent, due to the large amount of unfilterable char that remained in the oil. The time allowed to permit the suspended solids to settle was varied, but no decolorization was apparent after settling.

Fuller's Earth on heating "boiled" violently, due to the liberation of gases, but in the bleaching tests the results indicating that these gases were not necessary for the bleaching quality of the earth. The fact that the wetted sample of Fuller's Earth, later dried, gave better results than the raw, indicates that if the gases absorbed were CO_2, the water assisted in eliminating them by dissolving some and being driven off on heating, that CO_2 is not an assist to the bleaching properties of Fuller's Earth.

Studies of effect of acidity or alkalinity were made on the Georgia clay, because the preliminary study showed that no odor was left in the oil when this clay was used, while the oil treated with Fuller's Earth retained a distinct odor, or rather an odor was imparted to it. It is evident that the less the hydrochloric acid treatment, the better the grade of product, but that the same relationship holds true about causticity. It is apparent that the hydrogen-ion concentration of the bleach substance and the oil must be well regulated within certain limits and the bleach may be slightly acid.

The following conclusions are based upon the assumptions that holding the oil sample constant and the quantity of bleaching substance at 5% by weight, (*a*) that there is a better bleaching action with a slight acidity than with slight basicity, but that stronger acidity or basicity decreases this property; (*b*) that the Georgia clay used imparted less odor to the oil than the Fuller's Earth used; (*c*) that revivification of nearly all bleaches increase their bleaching power; (*d*) that the phenomenon seems to be a capillarity absorption one.

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THE EARLY HISTORY OF THE SOAP INDUSTRY

By L. W. Bosart

From almost the earliest times of which we have any record, use has been made of some addition to water to soften it or to increase its cleansing properties.

The early Greek writers, Aristophanes and Plato, who lived about 400 B.C., mention the use of an alkaline lye.

That the Phoenicians made use of soap has been claimed but has not been satisfactorily proven.

It is mentioned twice in the Old Testament. In Jeremiah II, 22, we read: "For though thou wash thee with nitre and take thee much sope." The Hebrew word "borith," which is translated "sope," probably means potash lye. In Malachi III, 2, reference is made to "fuller's sope." The Hebrew word "nether," which is here translated "sope," is believed to refer to a mineral lye, that is, to soda in some form.

Somewhat later we find an account of a soap, resembling in a measure soap as we know it today, given by the elder Pliny who died in 79 A.D. during the eruption of Vesuvius. Pliny gives some quaint methods of treating scrofulous sores, one of which is to use the burnt hoof of an ass or horse applied with oil or water. He then goes on to say: "Soap too is very useful for this purpose. This is an invention of the Gauls for giving a red color to the hair. It is made from tallow and ashes, the best from beechwood ashes and goats' tallow. There are two kinds, solid and liquid. Both of these are much used by the Germans, by the men in particular more than the women."[1]

It is said that the remains of a soap factory with soap in a state of perfect preservation was unearthed in Pompeii, and W. L. Carpenter, author of a book on soap making published in 1885, claimed to have some of the lime from it in his possession. From this, it would appear that the Pompeiians knew how to make caustic lye from soda or potash solution by treating with lime, and combined this lye with fat to make soap.

There must have been a great deal of soap in use at this time, but there is no evidence that it was used for purposes other than those mentioned by Pliny. Pliny's work "Historia Naturalis" is a comprehensive encyclopedia of the knowledge of his day. It seems quite improbable that he would not have made some mention of its remarkable cleansing properties, if they were known at that time. Further confirmation of this is the fact that nowhere does any mention seem to be made of the use of soap in the ancient, elaborate Roman baths.

It is a matter of some speculation how these people used it on the hair.

[1] "Prodest et sapo: Gallianum hoc inventum rutilandis capillis: fit ex sebo et cinere. Optimus fagino et caprino; duobus modis, spissus ac liquidus: uterque apud Germanos majore in usu viri quam feminis."

If they worked it into a suds, they would have to use water; if they smeared it into the hair, using water would be the easiest means of removing it afterwards. How they could have used water with soap and not learned of its action as a cleanser is difficult to comprehend.

In fact, one writer, Moride, is of the opinion that what Pliny meant to say was that the Gauls merely discovered the use of soap as a hair dye, and believes the real inventors were the Phoenicians who brought their art to Gaul when they settled at the mouth of the Rhone 600 years before Christ. But it is rather difficult to read this meaning in the Latin text.

The earliest writer to mention soap as a cleansing agent is Claudius Galen, who was born in Pergamus, Mysia, Asia Minor, 130 A.D. He spent much time in Rome and became renowned as a physician. Galen states that soap is made from fat and lye (from ashes with lime) and that it acts medicinally by softening and removing the dirt from the body and clothes.

It has been suggested that soap probably originated from the accidental mixing of oil and ashes, the mixture being used as a salve for skin eruptions and similar ailments. Later it was found by chance, perhaps, that a much more powerfully acting salve could be obtained if the ashes were first mixed with water and burnt lime and then with fat or oil. Gradually such a material was attained as is known today in the interior of Algeria and which serves both for cleansing and remedial purposes. It is a yellowish, somewhat transparent soap of the consistency of jelly and contains very little water. It is made of olive oil and caustic lye obtained from wood ashes and burnt lime.

Before the general use of soap for cleansing, the ancients are said to have employed the juices of certain plants, besides wood ashes and natural soda.

They also made use of fuller's earth. This was placed on the clothing and stamped in with the feet. The grease was absorbed by the earth and removed with it on scouring. Fuller's earth is said to have been employed also as a cleansing material in bathing and to have been used by the people of Rome as late as the 18th century.

It is probable that in the earlier centuries of the Christian era the Arabs and related people knew both soap and soda, as is indicated in the Koran.

The natives of West Central Africa are said to have made a kind of soap from palm oil and the ashes of the banana and plantain.

Strange to say the Chinese, who had the earliest knowledge of so many useful inventions, appear not to have known the use of soap until comparatively recent years when it was brought in to them from Europe.

The manufacture of soap underwent very slow development for several centuries after the time of Galen, but it seems to have flourished in Italy and Spain in the 8th century, where the olive furnished the bulk of the fat used. No really great advance was made until the early part of the 19th century when the researches of Chevreul into the constitution of fatty

bodies and Leblanc's discovery of how to make soda from salt, about the year 1790, opened the way for the establishment of the soap industry on a scientific and practical basis. These two men may therefore be regarded as the founders of the soap industry. In the 13th century Marseilles became famous as a soap center especially for the manufacture of olive oil soap. Other Mediterranean coast cities, Genoa, Savona and Venice became lively competitors of Marseilles and for several centuries kept up an active trade, but Marseilles has long upheld its preëminence. Besides the name Castile, indicating its Spanish origin, olive oil soap has come to be known also as Marseilles soap.

It was quite natural that the soap industry—such as it was—should settle about Marseilles. The olive tree with its oily fruit flourished in the South of France; moreover it was a seaport town so that it was in easy touch with the olive districts of the Mediterranean. Sea plants were close at hand which on being burned gave barilla, a crude soda which was used as a base for making hard soaps, while neighboring forests furnished wood from the ashes of which potash for soft soap could be obtained.

Soap does not appear to have been known in England at all until the 14th century. The first record of its manufacture there was in 1524. In 1622 an English soap company was granted a monopoly for the manufacture of soap, paying a yearly tax of 20,000 pounds for 3000 tons.

A monopoly was also formed in France in the 17th century.

Even at this early date there were complaints about adulteration and in 1688 the French government laid down definite rules for its manufacture, allowing the use of only barilla, soda, ashes and olive oil, without other fat or other material. But this was not entirely satisfactory and these rules were later revoked and with the outbreak of the French Revolution withdrawn entirely.

Two factors which developed about the beginning of the 19th century were of the greatest importance for the progress of the soap industry. One of these was the investigation of Chevreul into the constitution of fats, which gave to science the first actual knowledge of the chemical nature of fats and oils and consequently of soap; the other was the discovery of a means of making soda from common salt.

The soap industry was dependent for its existence on an adequate supply of soda or potash. It is probable that natural soda or natron was early brought into Italy from some of the Mediterranean coast countries of Asia and Africa, for considerable quantities of soda must have been available to supply the large glass industries of Italy. Certain it is, however, that wood ashes and barilla (the ash of seashore and sea plants) were the greatest sources of supply of alkali for soap making until about a century ago.

In 1775 the French Academy of Science offered a prize of 2400 pounds for a satisfactory method of making soda from salt.

De la Méthérie proposed to ignite sodium sulfate, made from salt and oil of vitrol, with charcoal. But the product of this reaction was principally sodium sulfide.

This, however, suggested the solution of the problem to Nicolas Leblanc, physician to the Duke of Orleans. His thought was that the addition of chalk or limestone would cause the formation of soda. Jean D'Arcet, professor of chemistry of the College of France, tried it out in his laboratory and made a favorable report. The Duke of Orleans advanced 200,000 francs to exploit the process and a factory was erected at St. Denis near Paris for the purpose. This was in successful operation for several years and produced from 500 to 600 pounds of soda ash per day. Then reverses came. As a result of the Revolution in France the Duke met his death on the guillotine, the plant at St. Denis was confiscated, the supplies sold and Leblanc was forced to reveal the secrets of his process for the good of the commonwealth. Leblanc was ruined. In 1800, by way of compensation, his factory was returned to him, but he was unable to get financial assistance and finally in 1806 in poverty and despair he took his own life. The prize which he had fairly won was never paid him. Eighty years later a monument was erected to his memory in Paris.

Fortunately his discovery lived on, but was exploited to a much greater extent in England than in France. It was not until about 1824 that James Muspratt began the manufacture of soda on a large scale in England. At first there was a certain prejudice of soap makers in favor of barilla to be overcome, and it is said that he had to give away this much purer product by the ton before he could get them to use it and to realize that it was actually a better and more economical material.

It would be difficult to overrate the value of Leblanc's discovery both to chemical industry and to the world's civilization. Certainly without it there would have been no great soap industry. The modern glass industry is also dependent upon the artificial production of soda. Great quantities of sulfuric acid were necessary to convert salt into sodium sulfate. This acid, which is essential to so many chemical industries, was therefore produced on a greater scale and at a lower price. Hydrochloric acid, chlorine and sulfur were valuable by-products which played a part in the development of other chemical industries.

The Leblanc process has served its purpose. That it has been almost entirely displaced by the ammonia-soda process of Solvay, does not in any way detract from the value of the part it played in the world's progress and industrial development.

In the early days of this country soap making was practised as a household art. The ashes from burnt wood were saved and leached out with water. This solution was boiled with the kitchen fats or the suet from slaughtered animals to produce a crude form of soap. Early in the 19th

century the manufacture of soap began to develop into a separate industry. Manufacturing plants were established in most of the larger centers of population. The industry has been marked by a continuous and rapid growth and today about two and one-half billion pounds of soap are produced annually in the United States.

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ABSTRACTS FROM OTHER JOURNALS

Glycerides of palm-kernel oil. A. BOMER AND K. SCHNEIDER. *Z. Unters Nahr. Genussm.* **47,** 61–89(1924); through *J. S. C. I.* **43,** No. 23.—Five almost pure mixed glycerides were isolated from a sample of palm-kernel oil by repeated fractional crystallization from acetone. The fatty acids in these glycerides were separated and subjected to steam distillation in Polenske's apparatus. The nature of the volatile fatty acids was investigated by determining the neutralization number of a saturated aqueous solution of the acids and by the analysis of the silver salts of the acids. The solid saturated fatty acids were separated from the unsaturated acids by Farnsteiner's method, and were fractionally precipitated with barium acetate solution by Heintz's method, the acid value and molecular weight of the acid obtained from each fraction being determined. By these methods the five glycerides were identified as caprylo myristo-olien (saponif. value 243.2 m. p. 13.9°) myristodilaurin (saponif. value 253.1, m. p. 33.4°), laurodimyristin (saponif. value 242.5 m. p. 40.0°), palmitodimyristin (saponif. value 227.5, m. p. 45.2°), and myristodipalmitin (saponif. value 216.0, m. p. 51.4°). The first two of these glycerides were present in considerable amount, the third only in small amount, and the last two in very small amount, comprising the most difficultly soluble glycerides in the oil. It is very unlikely that stearic acid is present in the glycerides of palm-kernel oil and caproic acid was not present in the sample investigated. It is uncertain whether capric acid was present or not. The glycerides present in coconut and palm-kernel oils are compared with one another in an appendix. H. C. R.

Uncommon animal fats. W. N. RAE. *Analyst* **49,** 83–84(1924); through *J. S. C. I.* **43,** No. 14.—The analytical characters of fats from the following wild animals of Ceylon are given: leopard (Felis pardus), sambhur (Cervus unicor), and wild boar (Sus crustatus). H. C. R.

	d^{99} 15	Butyro-refractometer reading at 40°, of: Fat.	Fatty acids	Acid value	Saponif. value	Iodine value (Wijs)	Unsaponifiable matter, %	Solidif. pt. of fatty acids	Neutralization value of fatty acids	Iodine val. (Wijs) of fatty acids
Leopard	0.8592	49.5	38.9	1.78	201.1	62.2	0.33	39°.2	210.3	60.9
Sambhur	.8610	45.7	31.6	4.37	210.6	22.4	.56	48°.2	213.1	21.4
Spotted deer	.8640	44.3	31.6	4.98	212.3	23.8	.52	47°.8	215.6	22.65
Peafowl	.8947	54.5	46.9	8.70	215.3	66.4	.34	36°.4	216.6	66.7
Wild boar	.8851	52.0	..	11.12	216.4	44.8	.40	...	...	..

Tung oil. A. EIBNER, O. MERZ AND H. MUNZERT. *Chem. Umschau* **31,** 69–82 (1924); through *J. S. C. I.* **43,** No. 23.—A summary is given of the results of the more recent work on the constitution of tung oil, 75% of which is comprised of α-elaeostearic triglyceride, which changes under the influence of light into the isomeride, β-elaeostearic triglyceride. Both the α- and the β-triglycerides increase in weight at about the same rate when spread in thin films on glass plates and exposed to the air. After standing for 10 months in a closed brown glass bottle β-elaeostearic became insoluble in acetone and no longer melted, but charred at about 265°. . . . . The conversion of the α- into the β-acid, therefore, seems to play very little part in the mechanism of the drying of tung oil. The clouding of films of tung oil on drying is not in any way connected with isomerism, but is due to the formation of microscopic folds in the film owing to the changes in volume which occur during drying. Tung oil shows a much slower increase in acid value on drying than either linseed or poppy seed oils, and is less influenced by the humidity of the atmosphere during drying than is linseed oil. . . . . It is therefore considered that the drying of tung oil is to be looked upon rather as a colloidal phenomenon involving transformation from the sol to the gel form than as a case of molecular polymerization. H. C. R.

Determination of iodine values. I. Comparative investigations on the iodine values of fats. S. SCHMIDT-NIELSEN AND A. W. OWE. *Videnskapsselskapets Skrifter* 1923, I., *Mat. naturv. Klasse* (15) 77 pages; through *J. S. C. I.* **43,** No. 15.—Comparative investigations on non-drying, semi-drying and drying fats or oils were made to test the various methods for the determination of the iodine value. The experimental error in the tests did not exceed 0.05%. Where accurate results, which are reproducible and correspond to the true iodine value, are required, Hubl's method is the best, but the results must be corrected by means of the formula: $\frac{127f}{1=100i}\ (b-a)-(a/b_t)^2(b-b_t)$, where I is the iodine value, f is the factor of the thiosulphate solution, a is the thiosul-

phate consumed in the test, and *b* and *bt* are the quantities of the thiosulphate consumed in blank tests made at the beginning and end respectively of the determination proper. Using an excess of halogen of 80% a reaction time of 12 hrs. is usually sufficient, but a reaction time of 24 hrs. is preferable. Waller's process is unsatisfactory. Wijs' method is inaccurate owing to substitution if there is any excess of chlorine present; a small excess of iodine is to be preferred. Comparative results can, however, be obtained if for 0.2 g. of fat with iodine value less than 160. The reaction mixture is allowed to stand for 2 hours, for higher iodine values a period of 6 hrs. is necessary. The results are within 1.5% of the true iodine value. The reagent is best prepared by passing chlorine into a solution of iodine in glacial acetic acid until the color changes from dark brown to pale brown, and then adding a few crystals of iodine. The Hanus method is rapid and gives consistent results when a constant weight of fat is used, and can be recommended for industrial use. The great volatility of bromine and the sensitiveness of the reaction to light detract from the value of Winkler's method, which, however, if carried out in a dark room in red light gives good results. T. S. W.

Discoloration in corn (Maize) oil soap and the possibility of preventing its development. E. G. R. ARDAGH, J. C. BELL, F. S. SPENSE AND A. MURRAY. *Canadian Chem. and Met.* **7,** 302–7(1923); **8,** 7–11(1924); through *J. S. C. I.* **43,** No. 12.—The discoloration that develops in time on the surface of soap made from maize oil is not due to the color of the oil nor to sunlight, but results entirely from atmospheric oxidation of the saponified unsaturated glycerides, *e. g.*, sodium oleate and linolate. It is impossible to make a soap from ordinary maize oil that will remain white for any length of time when exposed to the air. Even when made in an atmosphere of nitrogen the resulting soap will in time turn brown in air. Overheating of the soap during manufacture greatly aggravates the defect. Dry soap, *i. e.*, a well-grained soap or one that has been smoothed and then dried, does not become discolored so quickly as a smoothed or moist soap. The more rapid deterioration of wet samples appears to be due to the sodium hydroxide set free by hydrolysis, which acts as a catalyst of the oxidation. The presence of sodium chloride appears to delay the development of color, probably because it inhibits hydrolysis. Even first class white commercial soaps made from maize oil will turn brown in a few hours when moistened and heated at 110°. If maize oil is very carefully heated in an inert atmosphere so that no local heating above 285° occurs, the resulting oil, in its iodine value has been reduced to 113 or less, will give a soap that resists oxidation and will remain white much longer than soap made from unheated oil. Soap made with a weak lye (10° B., d. 1 074) retains its whiteness much longer than that made with a lye of 18° B. (d. 1.143). H. C. R.

A neglected chapter in chemistry; the fats. E. F. ARMSTRONG AND J. ALLAN. *J. S. C. I.* **43,** 207–11T.—This article constitutes a valuable summary of our present knowledge of fats. The author very sensibly uses the term "fats" as covering both solid fats and liquid oils. It avoids confusion often present in the lay mind, resulting from the use of the term "oil" for mineral and essential oils.

Die Glyceride des Schweinefettes. I. Analytischer Teil. II. Synthetischer Teil. CONRAD AMBERGER AND ADOLPH WIESEHAN. *Z. Untersuch. fur Nahr. u. Genuss.* **46,** No. 5, 276.—Four samples of lard were investigated. Fatty acids isolated were oleic, palmitic, and stearic only. No linolic, lauric, or myristic acids found. Quantitative separation of one sample gave, oleic acid 60.0%, palmitic acid 32.2%, and stearic acid 7.8%. The authors consider these present almost wholly as mixed glycerides. The following glycerides were found: palmito-di-stearin, stearo-di-palmitin, oleo-di-stearin, m. p. 42°, oleo-palmito-stearin, m. p. 41°, palmito-di-oelin, liquid at room temperature, tri-olein probably present. Glycerides were prepared synthetically and compared with those isolated. The following structure is assigned to the glycerides isolated from lard: palmito-di-stearin is β-palmito-α,α-di-stearin; stearo-di-palmitin is β-stearo-α,α-di-palmitin; oleo-di-stearin is α-oleo-α-β-di-stearin; palmito-di-olein is α-palmito-α-β-di-olein; palmito oleo-stearin is (evidently) β-oleo,α-palmito-α-stearin. R. H. KERR

Beitrage zur Kenntnis der Glyceride der Fette u. Ole XI Die Glyceride des Palmkemfettes. A. BÖMER AND K. SCHNEIDER. *Z. Untersuch. fur Nahr. u. Genuss.* **47,** Nos. 1 and 2, 61.—Glycerides of palm kernel oil were separated by fractional crystallization and identified. Fatty acids isolated and identified. Capryllic, lauric, myristic, palmitic, and oleic acids were isolated and identified. Caproic and stearic acids were found not to be present, while the presence of capric acid remains doubtful. Glycerides found were caprylo-myristo-olein, m. p. 13.9°, α-myristo-di-laurin, m. p. 33.4°, α-lauro-di-myristin, m. p. 40.0°, α-palmito-di-myristin, m. p. (impure) 45.2° and α-myristo-di-palmitin, m. p. 51.4°. The first of these was present in large, the second in considerable, and the last three in small quantities. R. H. KERR

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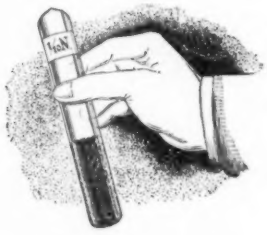

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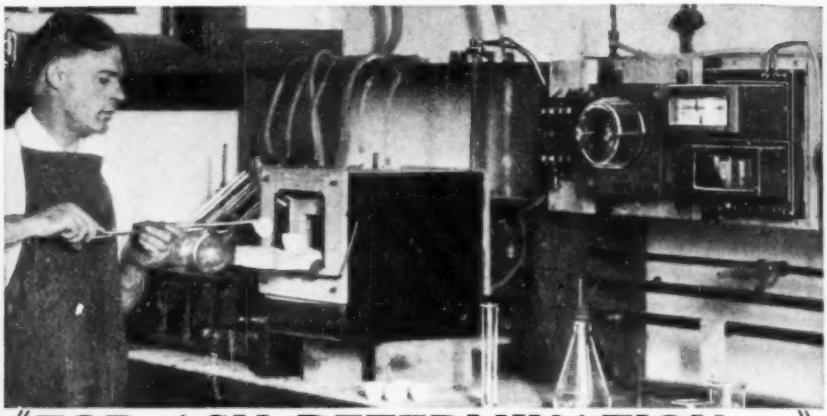

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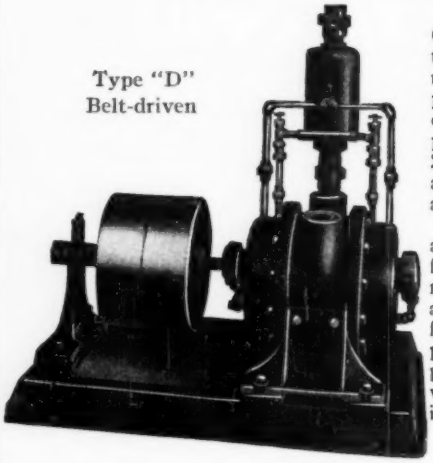

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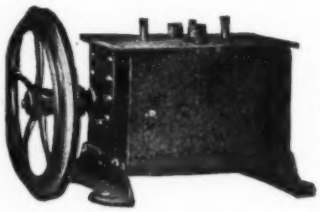

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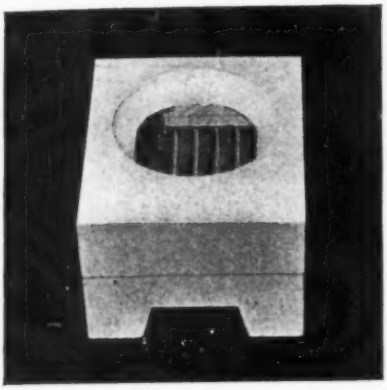

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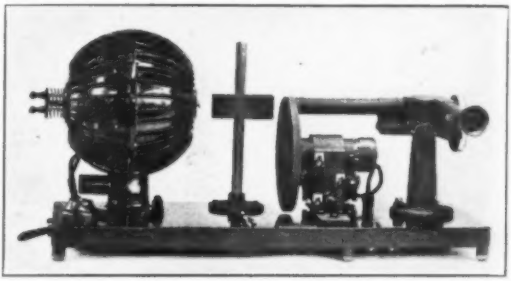

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